SAMPLE PREPARATION FOR X-RAY FLUORESCENCE ANALYSIS:

FUSING AND LAPPING

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1.0 PURPOSE

This procedure will describe the final step in the preparation of fused discs that will be analyzed for elemental composition in an x-ray fluorescence (XRF) analyzer. In this step, a powdered sample is transformed into a homogeneous glass disk. The fusing process utilizes a high temperature oven mounted on an orbital shaker table. High purity graphite crucible-molds containing a mixture of powdered sample and powdered lithiumtetaborate are placed in the oven and agitated while at a high temperature. The crucible- molds are then removed from the oven and allowed to cool. The resulting glass disk is then lapped to obtain a flat clean surface.

2.0 SCOPE

This procedure applies to fusing of powdered Yucca Mountain Project (YMP) geologic samples prepared for x-ray analysis.

3.0 PROCEDURE

3.1 Safety

3.1.1 The samples are fused only in a designated area on a metal topped table. The table has been equipped with splash guards.

3.1.2 Safety glasses must be worn during the performance of the procedure.

3.1.3 Hot crucible-molds are handled with tongs and the hot material must be kept over or on the metal table.

3.2 Cleanliness

3.2.1 The graphite crucible-molds are received from the weighing operation containing a mixture of flux and sample. Take care not to introduce foreign material into the sample. The final disk will be cleaned with alcohol, handled only by the sides and placed with the duplicate stored in a new ziploc plastic bag.

3.3 Traceability

3.3.1 Complete records shall be kept in the QA fusing logbook. The record will include; the fusion sample number, full sample name or number, date of preparation, the name of the investigator submitting the sample, and the signature of the person performing the fusion.

3.3.2 Fusion numbers will be engraved inside the bottoms of the graphite crucible-molds. When the fused disks are removed from the molds, the sample number will be permanently cast into the disk. Duplicates are made of each sample and are identified

with an a or b following the fusion number. Use extreme care when transferring sample identification as subsequent analyses rely on the correctness of the sample ID.

4.0 FUSING

4.1 Turn on the Linberg 59246 high temperature oven and set the control to 1150°C. Wait until the digital controller indicates that the oven has stabilized at the set temperature.

4.2 Place three or four empty crucibles in the Linberg high temperature oven and turn on the on/off switch.

4.2.1 Check that the temperature is set for 1150°C by pressing either of the two exposed buttons with white arrows on the left digital control panel. If a change is necessary, hold down the correct button until the desired temperature is reached (up arrow to increase the temperature; down arrow to decrease the temperature).

4.2.2 Check the following parameters by opening the small horizontal door on the portion of the left digital control panel. Depress the small black button and read each of the following parameters:

A1 = 1250°C	A2 = 0°C
Pb = 8	ti = 30
Td = 5	AP = 3
Hc = 0.3	HL = 100

If a change is necessary for any of these parameters, press the appropriate white arrow buttons (to increase press the up arrow; to decrease, press the down arrow). See operation manual for details (Eurotherm Corp.). Any change in these parameters will be recorded in the QA logbook.

4.3 Make sure that the samples are stirred thoroughly before putting them in the oven. Wipe the stirring rod clean between each sample.

4.4 Make sure the oven has reached 1150 C. Open the oven and using the long handle tongs place the crubible-molds in the oven.

4.5 Set the timer on the shaker table to twenty minutes and start the shaker action. The shaker should be set at between 1100 and 1300 rev/min.

4.6 At the end of the twenty minute time period remove the crucible-mold, look into the mix and if bubbles are present tip the crucible-mold slightly and tap it against the table top until the bubbles are removed.

4.7 Place the crucible-mold on a steel plate to cool to room temperature.

4.8 Remove the disk from the crucible-mold by turning upside down and tapping.

4.9 The fused disk is considered satisfactory if implementation of this procedure produces a homogeneous glass disk. If upon cooling the glass disk breaks, or if it contains excessive bubbles, the disk may be remelted in accordance with this procedure. It may be necessary to

increase the temperature or the time the sample is in the oven. If this is done, it must be recorded in the QA logbook.

5.0 LAPPING

5.1 Lap the flat opposite the cast fusion number.

5.2 Use the 100 micron diamond lap and flow water on the lap during the lapping operation. Sharp edges on the back of the disk may be removed if present. Next, repeat using the 30 micron diamond lap. Lap the disk until the entire surface is lapped and flat.

5.3 Place the disks in an ultra-sonic cleaner filled with alcohol. Place the disks, lapped surface down, on a brass wire mesh basket to assure that the disks are slightly above the bottom surface. Ultra-sonic for thirty seconds. Rinse with de-ionized water. Blow the lapped surface dry with a frozen duster (Do NOT use building compressed air).

5.4 Only those disks with smooth surfaces, lacking any visible surface irregularities (scratches, etc.) are acceptable.

6.0 QUALITY ASSURANCE

6.1 Personnel

Only persons certified to perform this procedure will perform this operation with YMP samples. Training for this procedure consists of reading the written detailed procedure and performing the procedure under the supervision of a trained person. The preparer of this detailed procedure is considered trained to perform this procedure and to train others. Evidence of training and certification shall be documented in accordance with the YMP Personnel Certification Procedure.

6.2 Calibration

Implementation of this procedure does not result in the production of data. The sample melting time, temperature, and shaker revolutions are not critical. Calibration of these parameters is not necessary.

6.3 Documentation

All samples fused shall be entered into a numbered QA logbook. The record will include fusion number, complete sample name or number, the name of the investigator, the signature of the person performing the fusion operation, the date, and comments the operator may have regarding unusual conditions noticed.

6.4 Responsibilities

The thin section lab supervisor is responsible to see that this procedure is followed correctly. This person is also responsible for the proper care and use of the equipment and to see that all calibrations are up to date. The thin section lab supervisor may delegate these responsibilities to a YMP certified person.

6.5 Accept/Reject Criteria

As stipulated in 4.9 and 5.4.

6.6 Storage

Upon completion, each glass disk, along with its duplicate, is to be kept in a separate zip-lock plastic bag. When samples are not in use, they are to be stored and locked in an appropriately labeled place.

There are no storage requirements for any equipment used in this procedure.

6.7 Procedural Deviations

Any deviations from this procedure shall be described in the fusing notebook and/or in the investigator's notebook.

6.8 Sample Transfer

Upon completion of this procedure, the disks are ready for X-Ray Fluorescence analysis. These finished disks shall be transferred to the XRF lab. There they shall be entered into a QA logbook and locked in a separate cabinet designated for YMP samples only until they are ready for analysis. This logbook shall include fusion number, complete sample ID, name of the investigator, name of the person making the entry, and the date of the entry. All entries shall be signed.

7.0 REFERENCES

Eurotherm Corp, Operation and Maintenance Manual for Microprocessor Based 3 Mode Controller, Model 810, Eurotherm Corp., 11485 Senset Hills Road, Reston, VA 22090.